METHODOLOGY

The SAXS intensity as a function of the scattering vector is due to the distance correlations of all the atoms in the particles of interest. Anomalous small angle X-ray scattering (ASAXS) refers to extensions of standard SAXS experiments in which the energy of the probing X-rays are tuned near the absorption edge of an element in the sample. By performing SAXS experiments near the characteristic absorption edge of any given atom, it is possible to vary the contrast for scattering of that particular element. This systematic variation in contrast yields the partial scattering functions of the specific atomic species. In general, the atomic scattering can be expressed as:

$$f(q,E) = f_0(q) + f'(q,E) + if''(q,E) \quad (1)$$

where E is the energy of the probing X-rays and q is the momentum transfer ($q=4\pi\sin\theta/\lambda$, where 2θ is the scattering angle and λ is the wavelength of X-rays). The parameters f' and f'' are the real and imaginary parts of anomalous dispersion. They each vary sharply at energies within 10 eV of the absorption edge. The imaginary scattering factor, f'', represents the absorption of X-rays which results in photoemission of a core electron. Variation in f' is responsible for the change in contrast seen in ASAXS signals. Near the absorption edge of a given atom the scattering intensity, I, varies as a function of energy or wavelength (Equation 2).

$$I(q,\lambda) = I_o(q) + f'(\lambda)\, I_C(q,\lambda) + [f'^2(\lambda) + f''^2(\lambda)]\, I_R(q) \quad (2)$$

Here I_o represents the nonresonant, energy-independent scattering. The cross term, I_C, reflects scattering between the specific element of interest and the remainder of the material, while I_R corresponds to the distance correlations of just the resonant scatterers.

Since f' and f'' are sharply varying functions near the edge, these experiments require the highest possible energy resolution (of the order of $\Delta\lambda/\lambda=10^{-4}$) for the probing monochromatic X-rays. In these experiments we determine the small angle scattering using incoming X-rays with 4 to 5 different energies. All but one of these energies are near the absorption edge of the atom of interest. The last measurement, using X-rays whose energy is 150 eV below the edge, gives a direct measurement of the nonresonant scattering, I_o, since at this energy f' and f'' are effectively zero. From these sets of data, in principle one can obtain a set of 3 to 4 differential scattering data after the subtraction of I_o. If the SAXS data as a function of energy are placed on an absolute scale, one can then use f' and f'' values to obtain the partial structure factors, I_C and I_R, by least square analysis. Established analysis techniques such as Guinier analysis, Gaussian fit of peaks, and modeling will be used for the analysis of SAXS data as well as the partial structure factors from the ASAXS data.

ADVANTAGES OFFERED BY APS FOR SAXS AND ASAXS

The monochromator in beamline 12-ID at APS employs two Si(111) crystals which provide a highly monochromated beam at a fixed exit (height and angle). The energy range from the first harmonic falls in the range of 3 to 22 KeV. An inspection of Figure 1 clearly indicates that ASAXS experiments can be carried out for elements with an atomic number up to 42 (Mo) by using their Kα absorption edges. Between an energy range of 22 to 35KeV, one has to use the third harmonic reflections to access Kα X-rays. This is an important region which covers elements such as Rh, Pd, Ag, Cd and Sn. The energies of the L_{III} edges of these elements are quite small and hence cannot be easily studied due to the high absorption cross sections of low energy X-rays. The rest of the high Z elements can be studied by using the L_{III} edges.

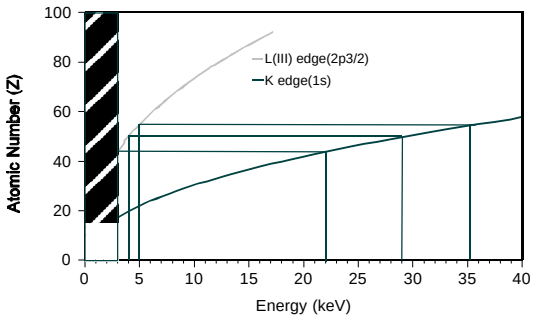

Figure 1. K and L_{III} absorption edges accessible for ASAXS measurements.

SAXS technique requires measurement of scattered X-rays in the lowest possible scattering angles as well as at a number of sample-to-detector distances to cover a wide range of scattering angles. ASAXS, in addition to the requirements for SAXS, also requires tunable X-rays in a wide energy range with high energy resolution. Both of these requirements are fulfilled by the high brilliance of the undulator beam line of APS. The small beam size and divergence at the BESSRC undulator beam line 12-ID will permit time resolved SAXS measurements in the sub-second time domain (down to milliseconds in favorable cases) and provide high energy resolution close to the Darwin width of the monochromator crystals for the ASAXS measurements.